CHEMISTRY 351

LABORATORY MANUAL

FALL 2010

FALL LABORATORY SESSIONS
START SEPTEMBER 13th 2010

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DEPARTMENT OF CHEMISTRY

UNIVERSITY OF CALGARY

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EXPERIMENTAL SCHEDULE FALL 2010

WEEK	DATES	NOTES	PRACTICAL WORK
1	Sept 13-17	**Check In & Orientation**	**1**: Solubility of Organic Compounds
2	Sept 20-24		**2.** Molecular Models
3	Sept 27-Oct 1		**3**: Melting point and Boiling point
4	Oct 4-8		**4**: Synthesis of Analgesics
5*	Oct 11-15	**Mon = THANKSGIVING***	**5**: Spectroscopy
6	Oct 18-22		**6**: Isolation of a Natural Product: Caffeine
7	Oct 25-29		**7**: Hydrocarbons and **8**: Chromatography
8	Nov 1-5	**MID-TERM WEEK**	**No laboratory**
9	Nov 8-12	**READING DAYS**	**No laboratory**
10	Nov 15-19		**9**: Hydrolysis of Sucrose
11	Nov 22-26		**10**: Reactivity in Substitution Reactions
12	Nov 29-Dec 3		**11**: Separation of an unknown mixture
13	Dec 6-10	**Check Out**	**11**: Separation of an unknown mixture

* **Students in laboratory sections scheduled for Monday, October 11th (Thanksgiving Day), will be required to make up the laboratory by attending another section that week.**

WHY ONLINE PDF ?

The CHEM 351 laboratory manual is available as a series of PDF documents. We don't expect you to print all the pages, some can just be read online (*e.g.* the background information), but you should print out the pages that describe the experimental techniques and the procedures and bring these to the laboratory sessions.

There are several sections to the manual documents:

Introduction

Techniques - experimental techniques are described in separate documents

Experiments - each experiment has at least two sections: background information and experimental procedure. In some cases, there is also a template for the written report.

1. OUTLINE

CHEM 351 is essentially the first half course of an introduction to modern organic and bio-organic chemistry. Since organic chemistry is a practically-oriented subject, the laboratory component forms an integral part of the course as it introduces basic laboratory techniques while investigating some of the modern concepts of structure, reactivity and reaction mechanisms of organic molecules in simple systems and in the often more complex biological context. Special attention has been paid to the selection of experiments in an attempt to clearly present and simplify some of the concepts of organic chemistry that students find difficult and to illustrate the rôle of organic chemicals in our everyday lives. We hope that one of the outcomes from the laboratory component of the course it that you learn more about the handling and management of chemicals.

In regard to the experiments, note that all the experiments **do work when carried out correctly and with good technique**. In general, they have been tried and tested over many, many years, so if the experiment doesn't work, it is quite likely due to experimenter error (that means YOU !).

In regard to the style of this manual, it is written in a scientific style and NOT as a "point form". You are taking a **University** science course and the procedures are written as you would find them if you were to go to a scientific journal or a standard laboratory text book. A "point form" style generally discourages students from thinking for themselves, something that is both very dangerous in a laboratory and very boring for the student.

2. LABORATORY COORDINATOR

If you have questions or issues with the laboratory component of the course, then you should contact the laboratory coordinator. While the laboratory TAs can help with questions about the

experimental work and the reports, it is the Laboratory Coordinator who is responsible for decisions about making up missed work, changing sections, exemptions, safety concerns *etc*.

Questions concerning the CHEM 351 laboratories in the Fall semester should be directed to the laboratory coordinator, Dr. Ashley Causton, room SA 144a, e-mail: acauston@ucalgary.ca

3. ATTENDANCE AT THE LABORATORY

Students are required to attend the laboratories each week and students are expected to be punctual for the laboratory sessions. **Students who arrive late** for a laboratory session will not be allowed to start the experiment and will be required to make up the laboratory in accordance with the information below. **The laboratory will close promptly at the end of the period since in many cases, the next section starts in 30 minutes,** so you will only be allowed 10 minutes "overtime" to tidy up.

4. MISSED LABORATORY SESSIONS

You are **required** to make up any excused absences *e.g.*, those for which an official medical note is provided to the Laboratory Coordinator. At the Laboratory Coordinators discretion, you will be allowed to make up other laboratory sessions that were missed for significant reasons. All absences must be made within **ONE WEEK** of returning to the university, ***and by prior arrangement*** with the Laboratory Coordinator. No absences can be made up once the same graded work has been returned to other students in the course.

In order to make up a missed laboratory section, you must **first** complete the "**Temporary Change of Laboratory Section**" form. The information on the Course Homepage will allow you to find another laboratory section **in your normal room** (so you can use your glassware) that you can attend. **Once completed**, have this form approved and signed by the **Laboratory coordinator** before you make up the experiment. **Once approved**, contact the Laboratory Technician (Ms. Cveta Hristoski SA 211, email: hristosk@ucalgary.ca) ***at least 24hrs in advance*** to enable the appropriate experiment to be set-up for you. **When you attend the make-up session**, you **must** get the TA in the section you attend to sign the form at the beginning of the laboratory period. This sheet **must** then be stapled to your report when you submit your report for grading.

Notes

If you try to make up an experiment without the having the form first approved by the Laboratory Coordinator, then the TA will **not** allow you to start work in the laboratory.

Any report submitted without the Temporary Change of Section form attached for an experiment that you missed in your regular section will **not** be graded.

5. GRADING

The Laboratory Course will account for 20% of the course mark of CHEM 351.

The laboratory course mark is based mainly on your laboratory reports but also on your preparation work, your notebook and the quizzes. It is important to think about the laboratory work and put it into context in terms of the big picture, the course as a whole. Look for the relations between the practical work and the other course components.

Use your time wisely…

It is important that students do well on examinations in order to get good grades in Chem 351. The examinations are the part of the course where we find out what an individual is capable of and it is the examinations in general, and especially the key performance indicator, the cumulative FINAL examination, that have the most significant influence on the course letter grade.

So for example if you spend 5 hrs working on a laboratory report worth say 1.5 course marks, then you should be spending about (40/1.5) x 5 hrs = 133 hrs preparing for the FINAL !

What does this mean ?

PUT THE MOST EFFORT WHERE IT IS MOST CRITICAL !

MOST STUDENTS...

- SPEND TOO MUCH TIME ON THE LABORATORY REPORTS AT THE ULTIMATE EXPENSE OF EXAMINATION PERFORMANCE.

- ARE MORE ANXIOUS ABOUT LABORATORY MARKS THAN THEY ARE ABOUT MAKING SURE THEY CAN DO WELL ON THE EXAMINATIONS.

In order to obtain a prerequisite pass the course (*i.e.* C- or better), a **minimum** passing mark in the Laboratory (50%) is **required**. Students are required to **complete a minimum of 7 of the 10 experiments (*i.e.* attend the laboratory session and submit the report). If a student misses the laboratory session, and then hands in a report, the report will not be graded. Students who fail to complete the minimum number of experiments (7 of 10) will be judged to have failed the laboratory component and therefore will be ineligible for a prerequisite pass in Chemistry 351. Therefore any student who has concerns about satisfying this requirement should talk to the laboratory coordinator as soon as possible, but NO LATER FRIDAY NOVEMBER 6th 2009.**

Laboratory reports will be graded based on the letter grade scale shown in the table below (for reference see University Calendar 2010-11, "Academic Standing: Grading System").

Note these letter grades are the **only** possible grades available for reports. If a report gets B/C it means it is slightly above the "C" average, but not good enough for a "B".

The grade of the reports will be assigned based on the overall QUALITY of whole report: your work and results, the scientific and technical content of the report and the answers to any

questions posed. The presentation, language, spelling and grammar used in your report are also important and will be used to evaluate the reports.

SEE THE LINK BELOW FOR FURTHER INFORMATION :

http://www.chem.ucalgary.ca/courses/351/laboratory/labgrades.html

For example, **a student who just adequately covers the minimum expectations in a report in an acceptable style will get a "C" letter grade - it's an average report !** General expectations are described below in section 4 and more specifically at the end of each experiment.

"Calendar" description	Letter grade	Marks earned
Excellent - superior performance	A	10
	A/B	9
Good - clearly above average	B	8
	B/C	7
Satisfactory - basic understanding	C	6
	C/D	5
Minimal Pass - marginal performance	D	4
Fail - unsatisfactory performance	F	2
Does not hand in report or does not attend laboratory section	--	0

The **Laboratory notebook** is important to all scientists and you will be required to maintain one for this course and it will be graded each week. For more details see the section "Laboratory notebooks". **Laboratory quizzes** will be given randomly throughout each course. These will be given at the beginning of the laboratory period under examination condition, and will be brief (5 minutes). Quizzes **must** be written in ink. The questions asked will refer to information in the laboratory manual or the references cited in relation to the experiment to be performed that week or lecture materials that relate to the experiment.

Overall the laboratory course components will be evaluated as follows:

CHEM 351 (150 marks max.)

Reports	= 110
Preparation	= 10
Notebook	= 10
Quizzes	= 20

If you disagree with any of the marks you have been awarded, you may appeal within **FIFTEEN DAYS** of **RECEIVING** the marks (as per the University Calendar 2010-11 "Reappraisal of Graded Term

Work"). The appeal should be made **first** to your laboratory instructor and **only then** to the laboratory coordinator. **If you need to appeal to the laboratory coordinator, then you need to provide the report and a written statement (clearly stating your concern) and your contact information within the 15 day period.** No such appeal will be entertained after the 15 days have elapsed. As per the University Calendar, "the reappraisal of term work may cause the grade to be raised, lowered or remain the same". For laboratory reports, since the grade is assigned to the whole report, the whole report must be regraded.

Each TA is provided with a marking guide for the reports, grading is discussed weekly at the TA meetings, and the grading is rotated so that different TAs grade the reports. Each of these factors help to standardise the grading process. However, due to the large number of laboratory sections and hence TAs for these courses, **the laboratory coordinator reserves the right to adjust (*i.e.* normalise) the marks for each section *if needed***. This is to ensure parity of marks for ***every student*** in the course.

6. PREPARING FOR THE LABORATORY

In order to be able to complete the experiments in the 3 hr. time slot available and in order to get the ***most*** from the experiments in CHEM 351, ***you*** will need to come to the laboratory ***effectively*** prepared. This means ***you*** should have read through the experiment, looked at the text references, and maybe the additional laboratory information on the course Homepage. This will allow you prepare for each experiment so ***you*** know what ***you*** will be doing, and will enable ***you*** to complete the experiments more efficiently and without making costly, silly, or even dangerous mistakes. Remember that each of the steps in the experiments is there for a *chemical* reason, and not simply to occupy your time. Try to think about these reasons and the procedures will all make more sense, and the experiments and the science will be more fun!

THEREFORE WE REQUITE THAT FOR THE PRE_LABORATORY WORK YOU :

In no more 4 or 5 sentences (about 100 words), identify the purpose of the experiment, what and how you will achieve that purpose, (*e.g.* what will you be measuring, doing, looking for *etc.*) and how you will establish how successful you have

You may also find it useful to write your own point form summary (or a plan) of what you are going to be doing - an example is provided (**see below**). Many students in the past have written out the whole procedure ahead of time - it's probably a waste of your time. It is what you **DO IN THE LABORATORY** that is important and needs to be recorded in the notebook as you perform the experiment. Your preparation will be evaluated and will form part of your laboratory mark.

7. LABORATORY NOTEBOOKS

A good laboratory notebook is fundamental to good science, so you are required to keep a notebook for CHEM 351. This will be graded. A self-duplicating laboratory notebook should be available from the University Bookstore.

The primary use for your notebook is to record **what you do as you do it** and observations **as you make them**. The notes should be detailed enough to allow another chemist of your level to pick up your notebook and repeat the experiment as you did it **without** the aid of this manual. Each experiment should start a new page that is dated and has the title of the experiment. Notebooks should be written in **ink** - **must not** be used). This information in your notebook can then be used to form the basis of your experimental report.

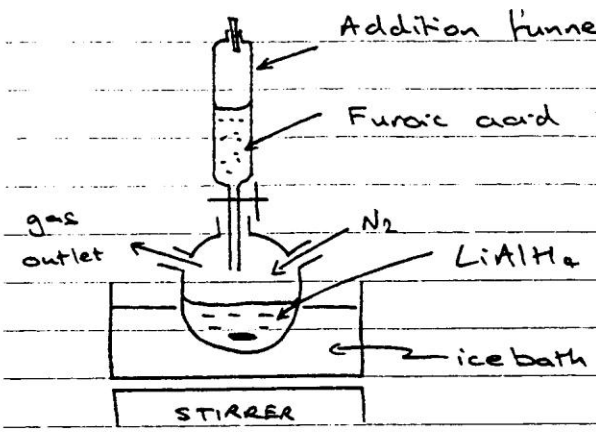
As indicated above, before each experiment you may wish to prepare a point form plan of what you will be doing based on the laboratory manual and other relevant materials. It is a good idea to list the properties of reagents, products, and solvents such as molecular weights, weights needed, molar quantities, melting points, boiling points, densities, and solubility properties beneath an equation that represents the preparative or kinetic experiment to be carried out. This information is easily expressed in table form and should be entered into the notebook before you come to the laboratory so it serves as an easy reference for the laboratory operations and helps you to minimise chemical waste. The "plan" may also include a sketch of any important apparatus, and should contain a stepwise analysis of the procedure to be carried out that can be referred to during the laboratory period. An example of a "plan" for a typical organic chemistry experiment is provided on the following page.

Once you are in the laboratory, the notebook should be used to record *all* your actual measurements (crude weights, purified weights, percentage yields, b.pts. m.pts. distillation temperatures *etc.*) and observations (colours of solutions, precipitates *etc.*) as you perform the experiment. Note any deviations from your point form summary. Separations are often easily described in flow-chart format. Difficulties encountered with laboratory operations and any accidental loss or contamination of product should be noted.

IMPORTANT: A duplicate copy of your notes **must be handed-in and your notebook graded and signed by laboratory instructor** before you leave the laboratory each week.

An example of a typical page from a laboratory notebook is provided as an example.

POINT FORM SUMMARY (SAMPLE)



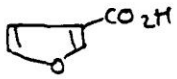
PROCEDURE

1. Dry glassware (100ml, 3 neck RBF)
2. Set-up for N_2 atmos + addition
3. Weigh $LiAlH_4$ (CARE!)
   @ 20 mmol = 0.76 g → into flask
4. Add 20 ml DRY diethyl ether
5. Cool to 0°C
6. Weigh acid
   @ 22 mmol = 2.47 g → funnel
7. 30 ml DRY diethyl ether → funnel
8. Add acid soln. slowly ~ 30 min.
9. Warm to RT, Ran overnight stir
10. WORK-UP
    0°C, add H_2O, NaOH, H_2O
    (.7 .7 2.1 ml)
11. Filter, remove solvent.

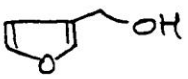
LABORATORY NOTES (SAMPLE)

Date: 16 Nov. 1992

Reduction of 3-furoic acid.



$LiAlH_4$



FW:	112.08	37.95	98.1
Eq:	1.1	1.0	
Used:	2.46 g	0.78 g	

$LiAlH_4$ (0.78g, 20.6 mmol) in dry ether (20ml) stirred at 0°C, N_2 atmosphere. Furoic acid (2.46g, 22 mmol) in dry ether (30ml) was added dropwise over ½ h. Cooling bath removed. Reaction was allowed to warm to room temp. and stirred overnight. After cooling back to 0°C, water (0.7 ml) was added dropwise, followed by 15% aq. NaOH (0.7ml) then water (2.1ml). The cooling bath was removed, and flask warmed to room temp. After filtering thro' Celite and washing with ether then methylene chloride, solvent was removed in vacuo to give an oil. The oil was distilled (12mm Hg, 85°) and gave 3-furyl methanol (1.75 g, 81%)

8. LABORATORY REPORTS

General Statement: We are more interested in **QUALITY** and not quantity. More is **NOT** necessarily better ! **In Science, it is important to be accurate, concise and to the point**. As a rough guideline, once you are used to writing reports, we would expect that an average student should be able to complete a reasonable write-up per week of experiment in an average of about 2 hours. If it is taking you longer you may be writing too much or you are not working effectively and you should reconsider how much time you are putting into the reports. The main reason that students do not do as well as they would like on reports is due to poor discussion of the scientific issues pertinent to the experiment

Generally, laboratory reports are due at the start **of the next laboratory period**, (that's usually **ONE WEEK AFTER** the experiment was carried out), typically in the next laboratory period. **It is your responsibility to make sure that your TA has your report by this deadline. Note that most of these reports are to be completed individually: copying and plagiarism will NOT be tolerated, and will be reported to the Department Head for appropriate disciplinary action.**

Any report not received by the TA by the deadline will be judged as "**LATE** d **WILL NOT BE ACCEPTED BY THE TA**. Late reports should be submitted except **to the Laboratory Coordinator of the CHEM 351 Laboratory Course** who will decide if there are acceptable extenuating circumstances.

There are two general formats for the CHEM 351 -out / fill-in-the-

The report is a formal description of your account of the experiment. As such it **must be written in a formal style and use proper English**. No slang or unacceptable abbreviations should be used (xs is not allowed, but *e.g.* is). If you are in any doubt which abbreviations are acceptable, then you should consult a dictionary. You should use the past passive tense as this tends to be a more readable style for the next person (or yourself) reading your report. **For example**, "2-bromobutane (2g) was dissolved in ethanol (25ml) and sodium iodide (2.5g) was added slowly**". The report should be written using sentences and paragraphs** and **NOT** in point form.

Reports can be word-processed or hand written in ink. If it word-processed, it should be line spaced at 1.5 and in Times (font size 12) or Arial (font size 10). For tear out reports many students try to cram in more material than is needed. There is enough space on the tear out pages to write what is needed in reasonable handwriting. If a report is illegible, needlessly untidy, uses very poor English spelling or grammar your grade will be affected.

The quality of what you write is important - in the future it may help you get a job - for example, a Calgary company has estimated that 25% of job applications go straight into the garbage because of the quality of the writing in the resume or the covering letter.

A pencil should only be used for drawing graphs and diagrams. All tables, figures, graphs diagrams etc. should be given a title.

A general format for a full laboratory report might include:

a) A brief descriptive **title** and the **date** the experiment was carried out and the names of any **co-workers**.

b) **Introduction** This briefly describes the purpose of the experiment (do not simply restate the title). In most cases, this will not be more than a few lines. An introduction should not contain any results!

c) Balanced **equation(s)** where appropriate. These summarise the chemical reactions and shows the basis for stoichiometric calculations.

d) **Procedure** A detailed description ***in your own words***, of the experiment that is sufficient to allow another chemist to repeat *your experiment*. Be sure to describe, in detail, any departures from the procedure in the manual *e.g*. time and temperature of reaction, spillages, or loss through leakage from a distillation assembly etc.

f) **Results and Discussion** Present your experimental results in a suitable format. Use tables and diagrams where appropriate and effective. Where appropriate, report the amount of product obtained both in grams and as a percentage of the theoretically expected yield. Record any physical properties you determine or observe and include literature values as a comparison (these values should be referenced - see below). If you measure a physical property more than once you must record all the values determined (*e.g.* for melting points). Record all your calculations. Include curly arrow mechanisms where appropriate. Discuss your results from a scientific basis.... for chemistry that probably means explaining things at the molecular level - what's happening and why.

g) **Sources of error** Describe any sources of loss or error etc. that have contributed to your experimental results. Describe any difficulties you encountered and any suggestions you have for improving the procedure. Assess your performance in carrying out the experimental operations. **Most students write far too much here** - essentially making things up and then write too little on the real science, the discussion and the conclusions. The most likely the principle sources of error will be because of what you have done.

h) **Conclusion** **Scientifically** assess your experiment and your results in general and record any deductions you have made. The conclusion should be a summary of how things turned out and will probably refer to points in your introduction.

h) **References**. Any claim you make must be supported by a reference indicating the source of your information. There are many ways to present references, but two common formats we recommend are illustrated below:

EITHER

p-nitrophenol by reduction with tin in glacial

and then at the end of the report, in the list of the references which are numbered in the order in which they are cited.

References

1. O. Morse, Ber., 11, 232 (1878).
2. A. Smith and B. Jones, J. Am. Chem. Soc., 99, 320-2 (l977)

etc.

OR

p-nitrophenol by reduction with tin in

and then at the end of the report, a list of the references, in alphabetical order based on the first-named author.

References

O. Morse, 1878, (title of the paper), Ber., 11, 232.

A. Smith and B. Jones, 1977, (title of the paper), J. Am. Chem. Soc., 99, 320-2

etc.

9. SAFETY AND WASTE MANAGEMENT

Safety is of the utmost concern in every chemical laboratory. The chemicals used in this laboratory may pose a variety of hazards. In order for students to be familiar with the safety issues related to working with chemicals in a chemistry laboratory, ALL STUDENTS are required to complete (if they atory , read the information provided here, and complete and pass the on-line safety assessment (in TopClass). If you have not previously taken the Department of Chemistry / Safety Office course AT THE UNIVERSITY OF CALGARY, YOU ARE **REQUIRED** TO DO SO WITHIN THE FIRST TWO WEEKS OF THE SEMESTER. This is a Federal requirement. Ask your TA or instructor for further details on how you can take the course. The on-line safety assessment is pass / fail. **Students who have not passed the safety assessment by 5pm September 24th 2010 will be suspended from the laboratory until they achieve a passing grade**. You will receive a zero for all work missed as a result of the suspension.

a) Regulations

The following regulations must be rigorously adhered to:

Students are **not** allowed in the laboratory unless an instructor is present.

Safety glasses with side shields **must be worn at all times** when in the laboratory.

Laboratory coats **must be worn at all times** when in the laboratory.

Shorts and sandals are **prohibited** in the laboratory.

All Food, beverages and smoking are **prohibited** in the laboratory.

Never pipette by mouth.

All unauthorized experiments are prohibited.

Pay attention to any hazards and warnings given in each experiment.

	If you see this symbol, you **must** talk to your TA before you continue. It means that there are significant hazards and that your TA needs to check before you continue.
	If you see this symbol, you should proceed with extra caution after checking your safety precautions. Check with your TA if in any doubt.

Experiments in progress **must not** be left unattended.

No overnight experiments are allowed.

Noxious or odorous chemicals are to be handled only in a fume hood. Generally, if you find a chemical in the fume hood, use it in the fume hood.

Wipe up spilled chemicals and bottle rings **immediately**.

Never handle or pour flammable liquids near an open flame or other ignition source.

Accidents must be reported **immediately** to the laboratory instructor.

At the end of each laboratory, **you are required to ensure your work area is clean and tidy**.

The safety form (available from your laboratory instructor) **must** be completed, signed, and turned into your laboratory instructor **before** beginning any laboratory work.

MSDS sheets are good sources of information concerning the hazards of a particular chemical, and are available for all chemicals used in the laboratory. The MSDS sheets are kept in blue binders at the TAs desk.

To avoid the possibility of chemical spills, you should keep your work area clean and free of clutter. In the event of accidental spillage inform your laboratory instructor immediately, and then clean up immediately. For larger spills, a spill kit is available in SA 211

In the event of accidental skin contact, ask a fellow student to inform your laboratory instructor while you immediately start to wash the area with copious amounts of cold water.

It is generally good practice to wash your hands frequently when working in any chemical laboratory.

In addition, you should learn the locations of the nearest telephone, fire extinguisher, eye wash station, emergency shower, and first aid kit, as well as exit routes from the laboratory and the building.

In case of emergency, the primary respondent should use the laboratory telephone and dial 220-5333 for assistance.

At the end of the laboratory period, make sure that the hood, work area and sink are clean and tidy.

Wash your hands before you leave.

b) Workplace Hazardous Materials Information System (WHMIS)

WHMIS provides that, by federal law, students are entitled to information concerning any materials used in the laboratory. This information is available on a Materials Safety Data Sheet (MSDS). For CHEM 351, these data sheets can be found in blue binders in each laboratory.

c) Safe Laboratory Practice

i) Fire

Know the location of the safety shower, the fire blanket and the fire extinguishers.

If the vapours from a flask ignite, a small fire can be extinguished by turning off the burner and gently placing a notebook or wire gauze over the top of the vessel containing the burning solvent. Under no circumstances move the flask. If it is necessary to use the fire extinguisher always take the fire extinguisher to the fire.

If solvent spilled on bench tops ignites, if possible, move bottles and flasks of unspilled solvent away from the area and then use the fire extinguisher. Never use the fire extinguisher wildly or more bottles may be knocked over and broken.

If your clothing is set on fire, move under the nearest safety shower and pull the chain.

ii) Burns

Beware of burns from hot glassware, hot iron rings or hot plates. Remember that the bulb of a thermometer in the melting point apparatus is at the temperature it records !

iii) Explosions

Explosions are very fast exothermic reactions and are usually given by substances which can undergo internal redox reactions e.g. polynitro compounds such as T.N.T. (trinitrotoluene), picric acid (trinitrophenol) or nitroglycerin, or by substances which can decompose to give much more stable compounds such as peroxides, azides, fulminates and diazo compounds. You will be warned if any particular explosion hazard exists with the compounds that you are using. One of the commonest causes of unexpected explosions is as a result of distilling solutions to dryness leaving behind traces of explosive residues such as peroxides. This is one reason why a distillation vessel is never heated to dryness.

iv) Poisoning, Skin Contact, Chemicals in the Eyes

Several of the compounds you will use are poisonous e.g. most alcohols, amines and nitriles. The regulations concerning eating and drinking in the laboratory **must** be strictly adhered to. Do not pipette liquids by mouth. You should always wash your hands before leaving the laboratory.

Certain harmful substances can be absorbed relatively easily through the skin e.g. dimethyl sulphate, nitrobenzene, aniline, phenol, and phenylhydrazine. Aromatic amines are serious carcinogens. Always minimise contact with any chemical and wash with water after accidental contact. You may wear latex surgical gloves, if you wish. These are available in the laboratory, or for improved protection, wear a pair of heavy duty gloves.

If harmful, irritating or flammable gases are used for or produced during a reaction, then that part of the experiment should be conducted in the fume-hood.

If you do splash something into your eyes wash them thoroughly (for about 15 minutes) by means of the water fountain. Contact lenses must be removed immediately in order to wash the eyes.

v) Mixing Chemicals

Extreme caution should be used when mixing certain chemicals. If in doubt ask your laboratory instructor. Be especially careful when dealing with waste as certain types of waste are incompatible (e.g. concentrated H_2SO_4 and organic waste).

Carefully observe the warnings about mixing water with certain reagents e.g. water should never be poured into concentrated H_2SO_4, or brought into contact with sodium. Chlorinated hydrocarbon solvent residues should be placed in the bottle provided. These should never be mixed with alkali. Never put concentrated H_2SO_4 in the organic waste containers. Specific warnings will be issued as required.

vi) Protection of Skin and Clothing

Acids and alkalis are extremely corrosive and will damage skin and clothing. Certain organic compounds are excellent dyes. This is the reason that you are required to wear a protective laboratory coat and safety glasses. Sandals or shorts should not be worn in the laboratory.

vii) Handling Reagents

Take great care to avoid contaminating reagents by:

Always replace bottle tops as soon as you have finished dispensing reagents since many compounds react with moisture in the air, with oxygen or with carbon dioxide. Others are quite volatile. Do not mix the tops. If you do remove the reagent from the common work area temporarily, replace it as soon as possible.

Always pour from the side of the bottle away from the label so that the name is not rendered unreadable. A reagent with no label at best is useless; at worst is dangerous.

viii) Tidiness

In order to work well, one of the first essentials is to keep your own, and the common areas such as reagent benches and fume-hoods, both clean and neat. Wipe up any spillages **immediately**. Care and tidiness in work is a large factor in safety, accuracy and efficiency.

d) Waste Disposal

This is a **VERY SERIOUS ISSUE**. When disposing of any waste material, **THINK** ABOUT WHAT YOU ARE DOING. This is for YOUR SAFETY and to PROTECT YOUR ENVIRONMENT. Failure to comply with the waste rules could lead to you being suspended from the laboratory sessions and hence the loss of laboratory marks.

Appropriate waste disposal is important, for example, chemically contaminated glass such as Pasteur pipettes cost \$1100 per m^3 as is buried whereas clean glass costs \$30 per m^3 and is recycled. **So, PLEASE MAKE SURE** the waste is going **into** the appropriate container. Here are some the rules:

SPECIFIC INSTRUCTIONS FOR THE DISPOSAL OF CHEMICAL WASTES ARE GIVEN AT THE END OF EACH EXPERIMENT.

NEVER POUR WASTE CHEMICALS DOWN THE SINK other than **very** dilute aqueous, non-toxic solutions.

ANY BROKEN GLASSWARE SHOULD BE COLLECTED INTO A BEAKER AND CLEANED (RINSED WITH SOLVENT AND THEN WATER) BEFORE TAKING IT TO THE TECHNICIAN FOR REPLACEMENT, WHEN IT IS THROWN AWAY in the BLUE PAILS provided. (Chemically contaminated waste is costly to dispose of and can pose an environmental hazard).

MAKE SURE WASTE IS PUT **IN** THE CORRECT CONTAINER

Broken glass or other sharp objects MUST BE **CLEANED** then placed in the BLUE PAILS specifically marked for this purpose.

Solid chemical waste should be placed in the container provided in the fumehood.

Organic solutions or solvents should be placed in the LARGE BLUE DRUMS in the fumehood.

Aqueous solutions should be placed in the aqueous waste container in the fumehood.

Sample vials should be placed in the special boxes provided.

Pasteur pipettes should be placed in the special container specifically for them.

"Non-chemical" solid wastes should be placed in the waste bins.

NEVER OVERFILL CONTAINERS (THERE **MUST BE** AT LEAST 5cm AIR SPACE AT THE TOP).

IF A CONTAINER IS FULL, THEN ASK FOR A REPLACEMENT.

CONSULT YOUR LABORATORY INSTRUCTOR IF YOU BREAK YOUR THERMOMETER (special precautions are required for disposing of mercury spills)

IF IN DOUBT, check your laboratory manual, and if you are still unclear, then ask your laboratory instructor or consult a copy of Aldrich Chemical Co. catalogue (this includes disposal information on all chemicals they sell) for appropriate procedures. The waste disposal area must be kept clean. If it is not, then expect your laboratory instructor to ask you to help clean it up.

10. CHECK IN / CHECK OUT PROCEDURES

DEPARTMENT OF CHEMISTRY BREAKAGE POLICY

Time has been allocated in the laboratory schedules for Check-In and Check-Out. During check-in you will be assigned a locker of organic chemistry equipment. A list of the equipment in your locker is included at the end of this manual. You are responsible for identifying the contents of your drawers (of course your TA will help you do this) and satisfying yourself that **all** the equipment listed on the equipment lists is **present**, **clean** and **useable**. The equipment should be complete, but if anything is missing it may be obtained from the laboratory technician in room SA211. If any glassware is chipped but is still usable (check with your TA if you are unsure), let your TA know. When you have determined that all equipment is **present**, **clean** and **useable**, you should sign the check-in form. **You are required to provide a lock in order to secure your locker**. We recommend a combination lock.

ONCE YOU SIGN THE CHECK-IN FORM, **YOU** ARE SAYING THAT THE EQUIPMENT KIT IS COMPLETE, CLEAN AND USABLE AND THAT YOU **ACCEPT RESPONSIBILTY** FOR ALL THE EQUIPMENT IN YOUR DRAWERS UNTIL CHECK-OUT. THERE WILL BE **ABSOLUTLEY NO** REPLACEMENTS ONCE YOU HAVE SIGNED THE FORM.

YOU ARE RESPONSIBLE FOR CLEANING IT, LOOKING AFTER IT, REMEMBERING TO PUT IT AWAY AFTER USE, AND SECURING YOUR DRAWERS.

The loss or breakage of any equipment assigned to you will result in you being charged for replacement of the item(s). The costs of replacement are included on the locker list (back of manual). If you break a piece of equipment or lose it, complete a replacement slip (available from your TA) then take it to SA211, where you will be issued with the replacement and the slip will be kept on file until check-out time when you will be billed for the replacements you have accumulated over the semester. You may also

be required to pay for the replacement of broken communal equipment (*i.e.* not part of the normal locker equipment) if the breakage resulted from careless or improper use on your part. The cost of communal items is $30.00 plus 60% of the remaining replacement cost.

It is important that you remember that you are also required to check-out of your locker by the end of each course **EVEN IF** you drop or withdraw. Please ensure that when you check-out everything is present and clean ! **FAILURE TO CHECK-OUT BY THE LAST WEEK OF THE LABORATORY COURSE WILL RESULT IN AN EXTRA $ 30.00 LATE CHECK-OUT FEE**, in addition to any appropriate replacement costs. Any charges will be collected at the end of each of the courses. You must ensure that you have paid any charges owing by the end of the examination period, or an additional $10.00 administration charge will be applied. *If you drop or withdraw from either course during the semester, you should check-out of your locker straight away before you forget.*

FAILURE TO PAY BREAKAGE / CHECK-OUT / LATE CHARGES WILL RESULT IN THE WITHHOLDING OF UNIVERSITY SERVICES.

11. USEFUL REFERENCES FOR PRACTICAL ORGANIC CHEMISTRY

The results of chemical and biochemical research usually appear as notes, communications, or papers in the various chemical and biochemical journals. The easiest access to this information is through one of the electronic databases.

However, if all the information you require is a melting point or a good recrystallisation solvent, a much simpler source of information would be one of the specialised secondary sources that have been developed. There are collections of methods of synthesis, methods of analysis, physical properties including melting point, boiling point, solubility, refractive index, vapour pressure, and also ultra-violet, infra-red, nuclear magnetic resonance (n.m.r.) and mass spectra, and many others.

Listed below are a few of the more useful references that you might need to consult.

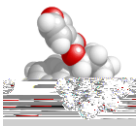
1. Handbook of Chemistry and Physics, 63rd edition, R.C. Weast, editor, Chemical Rubber Company, 1973. Contains physical and chemical properties for about 14,000 organic compounds, in addition to a host of other physical and chemical data. A copy of the "Rubber Handbook" is available in SA 211.
2. Dictionary of Organic Compounds, Heilbron, Oxford University Press.
   This dictionary is an alphabetical listing of over 25,000 organic compounds. It contains information concerning physical properties, recrystallisation solvents, reactions, derivatives, and literature references.
3. The Merck Index, 8th edition, Merck and Co. Inc., New Jersey, 1968
   This index lists about 10,000 compounds in alphabetical order and contains information concerning physical properties, solubility, hazards, and medicinal uses.
4. A Textbook of Practical Organic Chemistry Including Qualitative Organic Analysis, 3rd edition, A.I. Vogel, Longmans, 1961. An enormously useful volume which includes a detailed discussion of

the theory and techniques of practical organic chemistry, and detailed preparations of nearly 500 representative organic compounds.

You will also find the following laboratory texts available in the Reserve Reading Room in the University Library:

1. A Textbook of Practical Organic Chemistry, Vogel, 4th ed. Longmans, 1978.
2. Organic Experiments, Fieser & Williamson, 4th ed. D.C. Heath, 1979.
3. Introduction to Organic Laboratory Techniques, Pavia, Lampman & Kriz, Saunders, 2nd ed., 1982.

12. CHEM 351 Homepage



CHEM 351 maintains a Homepage on the WWW at:

http://www.chem.ucalgary.ca/courses/351

The Homepage can also be accessed via the University of Calgary homepage and following the

The CHEM 351 web pages contain information on all aspects of the courses, such as schedules, a weekly bulleti

publishers (McGraw-Hill) On-Line Learning Center for the Carey text book, plus links to other potentially useful WWW sites etc.

Within the Homepage is a section devoted to the laboratory that includes information, colour pictures and/or movies of important laboratory techniques and experimental set-ups, plus a few details of each weeks experiment. This material is designed to help you prepare for each experiment ahead of your normal laboratory period.

13. TA OFFICE HOURS

In order to provide more general support for the laboratory in a safer environment, the CAL facility, SA204, will be used. Therefore if you need to talk to a TA about your laboratory work or your report you should go there. This room is open in the afternoons Monday - Friday 12-4pm. The instructors in that room will be able to help you with your laboratory related questions, however, you should not expect them just to tell you the answers to your questions, they will try to encourage you and guide your thinking process. They will expect you to have thought about things yourself first - if you haven't don't expect them to help you and don't complain about it !

14. INTRODUCTION TO THE EXPERIMENTS

The CHEM 351 laboratory course provides:

An introduction to the various essential techniques of practical organic chemistry,

An introduction to the fundamental characteristics of organic molecules, particularly those of biological and industrial significance,

The opportunity to develop the skills required to work with the course material in general (*e.g.* spectroscopy, stereochemical analysis).

Chemistry is a practical subject. In particular, for an organic chemist much of the time, effort, and skills are devoted to the isolation of a product from a reaction mixture and its subsequent purification. The Chemistry 351 laboratories will introduce some of the important techniques for isolation, purification and characterisation while also investigating the reactivity of various systems in structure / reactivity correlations.

The practical materials in the manual are divided into two sections. The first part contains general information on the important experimental techniques and the second part contains the actual experiments. You will need to refer back to the techniques in order to complete the experiments. Each of the experiments covers a particular "task". Some experiments are quite short and only require part of a laboratory period but others are longer. If you refer to the experimental schedule you will see that in some weeks you have more than one experiment to complete, some experiments cover more than one week.

Experiment #1 is an investigation of the solubility of organic compounds in common solvents to probe the important connection of structure and function.

Experiment #2 is a "dry" lab looking at how atoms affect molecular structure and shape.

Experiment #3 introduces a standard organic laboratory practices for characterising solids and liquids, melting point and boiling point measurement.

Experiment #4 you will prepare and purify by recrystallisation two very well known analgesics (pain killers), acetaminophen and aspirin.

Experiment #5 is concerned with the development of spectroscopic problem solving skills, helping you to learn to interpret spectra and solve the "puzzle".

Experiment #6 is concerned with the extraction and purification of the naturally occurring stimulant, caffeine from tea using standard isolation techniques: liquid-liquid extraction, recrystallisation and sublimation.

Experiment #7 is the first of two experiments about reactivity, in this case it is the reactivity of hydrocarbons is that is investigated.

Experiment #8 brings together some of the previous experiments in a chromatographic analysis of drugs.

Experiment #9 investigates the kinetics of the hydrolysis of the common carbohydrate sucrose.

Experiment #10 is the second reactivity experiment, it probes "Reactivity in Substitution Reactions" as applied to alkyl halides.

Experiment #11 will require that you use the skills you acquired during the semester to separate and characterise a mixture on two organic unknowns.